Plant and Soil **247:** 253–260, 2002.

Clinoptilolite zeolite and cellulose amendments to reduce ammonia volatilization in a calcareous sandy soil

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Received 13 March 2001. Accepted in revised form 25 June 2002

Key words: ammonia volatilization, clinoptilolite, microbial biomass, nitrogen fertilizers, organic amendment, soil mineral N

Abstract

Leaching of nitrate (NO_3^-) below the root zone and gaseous losses of nitrogen (N) such as ammonia (NH_3) volatilization, are major mechanisms of N loss from agricultural soils. New techniques to minimize such losses are needed to maximize N uptake efficiency and minimize production costs and the risk of potential N contamination of ground and surface waters. The effects of cellulose (C), clinoptilolite zeolite (CZ), or a combination of both (C+CZ) on NH_3 volatilization and N transformation in a calcareous Riviera fine sand (loamy, siliceous, hyperthermic, Arenic Glossaqualf) from a citrus grove were investigated in a laboratory incubation study. Ammonia volatilization from NH_4NO_3 (AN), $(NH_4)_2SO_4$(AS), and urea (U) applied at 200 mg N kg^{-1} soil decreased by 2.5-, 2.1- and 0.9-fold, respectively, with cellulose application at 15 g kg^{-1} and by 4.4-, 2.9- and 3.0-fold, respectively, with CZ application at 15 g kg^{-1} as compared with that from the respective sources without the amendments. Application of cellulose plus CZ (each at 15 g kg^{-1}) was the most effective in decreasing NH_3 volatilization. Application of cellulose increased the microbial biomass, which was responsible for immobilization of N, and thus decreased volatilization loss of NH_3–N. The effect of CZ, on the other hand, may be due to increased retention of NH_4 in the ion-exchange sites. The positive effect of interaction between cellulose and CZ amendment on microbial biomass was probably due to improved nutrient retention and availability to microorganisms in the soil. Thus, the amendments provide favorable conditions for microbial growth. These results indicate that soil amendment of CZ or CZ plus organic materials such as cellulose has great potential in reducing fertilizer N loss in sandy soils.

Introduction

For a mature orange (*Citrus sinensis* L. Osbeck) or grapefruit (*Citrus paradisi* MacFadyen) grove, 135–270 kg N ha^{-1} yr^{-1} are usually applied to sustain optimal yield and fruit quality (Tucker et al., 1995). Of the applied N, about 400 g kg^{-1} is accounted for in the harvested fruit, and the fate of the remaining 600 g kg^{-1} is not clear (Tucker et al., 1995). Soils in the major citrus growing areas in Florida are extremely sandy (often sand content >950 g kg^{-1}) and have a low nutrient retention capacity. In addition, around 30–40% of the soils under citrus are calcareous with pH above 7.5. High summer temperatures and excessive rainfall in Florida provide favorable conditions for nutrient loss through leaching and/or volatilization in the citrus production system.

Nitrogen applied as urea or NH_4^+ undergoes chemical transformation to produce either ammonia (NH_3) or nitrate (NO_3^-), depending on soil pH, moisture conditions, and application methods (Avnimelech and Laher, 1977; He et al., 1999; Nelson, 1982). In the

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past, extensive studies were conducted to quantify N leaching loss in these sandy soils (Calvert, 1975; McNeal et al., 1995; Paramasivam et al., 2001) because of public concern that NO_3^- that is not absorbed by the tree root system can be leached below the root zone and results in contamination of the groundwater (USEPA, 1987). However, by optimizing irrigation and avoiding fertilization during the rainy seasons (usually from mid-June to mid-October), leaching loss of N appeared to be limited (Paramasivam et al., 2001).

Dry application of water-soluble granular blend of fertilizers containing AS, AN or U using a spreader is the mainstay of fertilization for citrus production system in Florida although fertigation practice has increased in recent years. Application of dry fertilizers is generally conducted in February, May and late October during the dry season (generally from late October to early June next year). Dry soil condition, high soil temperature (particularly in May and October) and high soil pH during the dry season are favorable to the production of NH_3 from surface-applied NH_4^+–N or urea. Therefore, NH_3 volatilization may contribute substantially to the N loss in the calcareous sandy soils that cover a large proportion of the total citrus-growing area in Florida. Previous study showed that 150–240 g kg^{-1} of the applied N could be lost by volatilization in a Riviera fine sand (pH 7.9), depending on fertilizer sources (He et al., 1999). Ammonia volatilization may also result in contamination of lakes and streams, for the NH_3 volatilized into the atmosphere can be trapped by rainwater (Hutchinson and Viets, 1969; Sommer and Hutchinson, 1995). Re-deposition of NH_4^+ may also adversely affect land ecosystems by contributing to soil acidification and nutrient imbalances, etc. Therefore, it is important to identify new approaches to reduce NH_3 volatilization in an effort to maximize the N uptake efficiency by the citrus trees and to minimize the potential impact of agricultural activities on water quality.

Many factors influence NH_3 volatilization from soil. Among them are rate, source and method of nitrogen application, pH, temperature, cation exchange capacity (CEC), $CaCO_3$ content, and moisture content of soil, etc. (Fan and Mackenzie, 1993; Fenn and Kissel, 1974; He et al., 1999; Nelson, 1982). Ammonia volatilization generally increases with NH_4–N rate and decreases in the following order for different N sources: $CO(NH_2)_2 > (NH_4)_2SO_4 > NH_4NO_3$ (Gezgin and Bayrakll, 1995; He et al., 1999).

In soils with low CEC, ammonia volatilization can be minimized by appropriate sinks in the soil for NH_4–N. Clinoptilolite zeolite (CZ), a porous mineral with high cation exchange capacity (CEC, up to 3000 $mol_c\ m^{-3}$) and with great affinity for NH_4^+ (Ming and Mumpton, 1989), has been used to reduce NH_3 emission from farm manure (Amon et al., 1997), and to eliminate NH_3 toxicity to plants (Gupta et al., 1997). Ammonium retained by CZ is generally subjected to slow release through cation exchange and nitrification in soil (Kithome et al., 1998). Amendment of CZ to sandy soil has been reported to lower NO_3^- and NH_4^+ concentrations in the leachate and to increase moisture retention in the soil due to increased soil surface area and CEC (Huang and Petrovic, 1994). Application of $(NH_4)_2SO_4$ loaded into CZ was observed to minimize N leaching and to increase N utilization by crops in sandy soils compared with $(NH_4)_2SO_4$ alone (Perrin et al., 1998a,b).

Microbial biomass serves as both a driving force of N transformation and a potentially available N pool (Smith and Paul, 1991). Application of organic materials or N fertilizer can enhance the growth of microbial biomass (Wang and Bakken, 1997). Soil microbial biomass has an average C/N ratio of about 15 (Jensen, 1997). Soil amendment with organic materials with high C/N ratio reduces mineral N concentration in soil because of enhanced immobilization (Gallardo and Merino, 1998). Immobilized N due to increase in microbial biomass is subject to mineralization after the added C or N sources are depleted (Smith and Paul, 1991).

In Florida, there is increased concern over contamination of waters by overloading of nutrients from non-point sources. One of the objectives of the best management practices currently studied is to minimize N transport from land to waters. The objective of this study was to evaluate the effectiveness of cellulose and/or CZ amendments in reducing NH_3 volatilization in a calcareous sandy soil.

Materials and methods

A calcareous Riviera fine sand (loamy, siliceous, hyperthermic Arenic Glossaqualf) was sampled at 0–0.2 m from a commercial grapefruit grove in Martin County, FL. Some properties of the soil were: pH 7.9 at 1:1 soil/water ratio; sand 958, silt 28, and clay 14 g kg^{-1}; and organic matter 15.2 g kg^{-1}, cation exchange capacity 47 $mol_c\ m^{-3}$, and $CaCO_3$ 28.3 g kg^{-1}. The soil sample was air-dried and ground to pass through a 2.0 mm sieve. The zeolite used was clinoptilolite (CZ) from a natural source in Colorado. The CZ was ground to pass through a 0.074 mm sieve and had a CEC of 2100 mol_c m^{-3} with Na^+ as the dominant cation (pH 9 at 1:1 solid/water ratio, and 40 g kg^{-1} $CaCO_3$ equivalent). Pure cellulose (SIGMACELL type 101, Sigma Chemical Co., St. Louis, MO)[1] was used as a model organic material in this study.

Incubation study

A factorial design was used in a laboratory incubation study. Four N treatments consisted of: the control (without amendments), or 200 mg N kg^{-1} soil as either NH_4NO_3 (AN), $(NH_4)_2SO_4$ (AS) or urea (U). The N rate was higher than grovers' application rate (180–220 kg N ha^{-1}) on the consideration that fertilizers were surface applied during the dry season and most of the applied N was limited within 0–5 cm soil depth. Soil amendment treatments consisted of either unamended soil or application of 15 g kg^{-1} soil of either clinoptilolite zeolite (CZ) or cellulose, and CZ (15 g kg^{-1})+cellulose (15 g kg^{-1}). There were three replications for each treatment. Portions of air-dried soil were weighed and thoroughly mixed with the respective treatments. The total weight of each soil-amendment mixture was 0.4 kg (oven dry basis). The moisture content of the mixture was adjusted to 700 g kg^{-1} of field holding capacity.

The mixtures were placed in 500-mL plastic bottles with 45 mm ID opening with a screw cap (Rubbermaid, Inc., Wooster, OH) that features a short pour spout of 17 mm ID with an air-tight snap cap. A sponge-tracking and KCl extraction method was employed to measure NH_3 volatilization. This method was proposed by Cabrera et al. (1994) and modified by He et al. (1999) to measure NH_3 volatilization for laboratory incubation study with a reasonable accuracy and precision based on recovery and reproducibility tests. A more detailed description of the method development was given in He et al. (1999). Briefly, a sponge spiked with 1.0 mL of H_3PO_4–glycerol mixture (containing 35 mL of concentrated H_3PO_4 and 250 mL of glycerol per L) was inserted in the cap and the spout was left open to allow the NH_3 volatilized from the soil mixture in the bottle to be absorbed by the H_3PO_4–glycerol solution in the sponge. Each bottle was placed inside a sealed plastic Ziploc® storage bag (270×280 mm) to prevent contamination with NH_3 from the atmosphere and incubated at 30 °C. To simulate circulation conditions in the field, every morning, a gentle vacuum was applied to each individual bottle for about 30 s so that the air from inside the bottle could slowly pass through the sponge and NH_3 in the air could be absorbed by the phosphoric acid contained in the sponge. After incubation for 1, 7, 14, 21 or 28 days, the NH_3–N trapped in the sponge was extracted with 25 mL of 1.0 *M* KCl in a leak-proof plastic bag, which minimizes moisture loss and air contamination. A blank (empty bottle) in triplicates was also included for background correction, but concentration of NH_4^+–N in the blank samples was below detection limit (10 μg/L). Simultaneously, 2.5 g soil were taken in triplicate for extracting NH_4–N or NO_3–N using 25 mL of 1.0 *M* KCl shaken for 30 min and the incubation continued. The concentrations of NH_4–N or NO_3–N in the KCl extract were determined by a Cd-reduction rapid flow analyzer (RFA, method A303-S020, Alpkem Inc., College Station, 1989).

At the end of the incubation, moist soil samples were analyzed for microbial biomass using a fumigation–extraction method (Wu et al., 1990). Briefly, fresh moist soil samples (10 g oven-dry basis) were exposed to alcohol-free $CHCl_3$ vapor in a vacuum desiccator at room temperature for 24 h. The fumigated soils were then placed in a clean empty desiccator and residual $CHCl_3$ removed from the fumigated soil by repeated evacuation. Microbial biomass C was measured by extracting the fumigated soil immediately following $CHCl_3$ removal by shaking for 30 min with 40 mL of 0.5 *M* K_2SO_4 at solution: soil ratio of 4:1. After filtering (Whatman No 42 filter paper), the filtrate was analyzed for dissolved organic C using an automated TOC analyzer (5000 model, Simadzu Inc., Japan) and the biomass C was calculated from the increase in extractable C in the fumigated soil over that in the control (without fumigation) using a conversion factor (K_{EC}) of 0.45 (Wu et al., 1990).

Statistical analysis

The resulting volatilized NH_3–N, soil NO_3–N and NH_4–N concentrations, and soil microbial biomass from the different treatment combinations were analyzed using General Linear Model of SAS (SAS Inc., 1996).

* Mention of particular companies or commercial products does not imply recommendations or endorsement by the University of Florida over the other companies or products not mentioned.

Table 1. The volatilization loss of applied fertilizer N from a calcareous Riviera fine sand (N rate: 200 mg kg^{-1} soil, as either ammonium nitrate (AN), ammonium sulfate (AS), or urea (U)) during the 28-day incubation without additional amendments, or with either cellulose (C, 15 mg kg^{-1}) or clinoptilolite zeolite (CZ 15 mg kg^{-1}) or both (15 mg C+15 mg CZ kg^{-1} soil)

Treatments	AN (g kg^{-1})	AS (g kg^{-1})	U (g kg^{-1})
Control	94a[a]	125a	239a
C (15 mg kg^{-1})	27b	40b	125b
CZ (15 mg kg^{-1})	17b	32b	60c
C (15 mg kg^{-1})+ CZ (15 mg kg^{-1})	3.0c	6.0c	29d

[a]Different letter after each mean value within the same column indicates significance at 5% level by Duncan's Multiple Range Test.

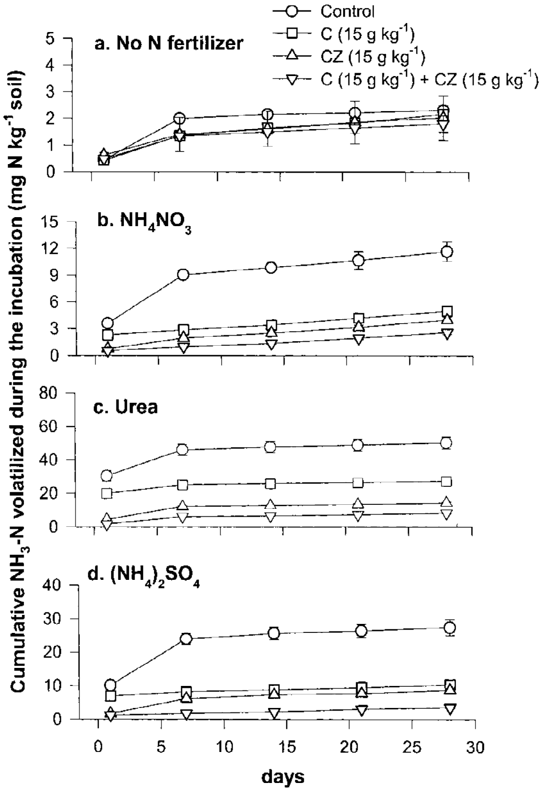

Figure 1. Cumulative NH_3 volatilization from a Riviera fine sandy soil with application of different nitrogen sources during the incubation (C=cellulose, CZ=clinoptilolite zeolite, error bar=standard error).

Results

Ammonia volatilization

Ammonia volatilization from the unfertilized soil was minimal and there was no significant difference in NH_3 loss among the various treatments (Figure 1a). Much more NH_3 was volatilized from the soil receiving 200 mg N kg^{-1} soil as AN, AS, or U, as compared with the control (Figure 1b–d). Amendment of cellulose or CZ significantly reduced NH_3 volatilization from the fertilized soil, and the CZ appeared to be more effective than cellulose, especially for the urea treatment. Application of cellulose plus CZ resulted in the least NH_3 volatilization from all N sources (Figure 1b–d and Table 1).

Dynamics of ammonium-N and nitrate-N in soil

The concentration of NH_4–N in the soil without N addition or with 200 mg N kg^{-1} as AN or AS decreased over the incubation time (Figure 2). In the soil received 200 mg N kg^{-1} as U, the NH_4–N concentration increased in the first week of incubation, as a result of NH_4–N release from urea hydrolysis, and then rapidly decreased. The decrease in soil NH_4–N concentration could be attributed to NH_3 volatilization (Figure 1), microbial immobilization, and nitrification as discussed later. Cellulose addition decreased more the NH_4–N concentration in 3 weeks of incubation, as compared with the control or CZ treatment (Figure 2), probably because of an enhanced microbial immobilization. Clinoptilolite zeolite (CZ) appeared to maintain relatively higher NH_4–N concentrations in the soil receiving either U or AS compared to the control or cellulose treatment, probably due to retention of NH_4 on cation-exchange sites of the CZ, which reduced NH_3 volatilization (Figure 1). For the soil receiving AN, the measured NH_4–N concentrations in the CZ-amended soil were slightly lower than the control, which was probably due to incomplete recovery of NH_4–N in the CZ-amended soil by the single KCl extraction procedure. However, the difference in NH_4–N concentration among the various amendment treatments was not significant for all sampling dates, and the NH_4–N concentration was less than 50 mg kg^{-1}, 14 days after incubation across all N and soil amendment treatments (Figure 2).

Application of N fertilizer increased NO_3–N concentrations in the soils (Figure 3). In the soil without cellulose or CZ, concentrations of NO_3–N increased up to 14–21 days of incubation depending on the N sources, as a result of nitrification (Figure 3). Application of CZ decreased the NO_3–N concentration in the AN and AS treatments, probably because of

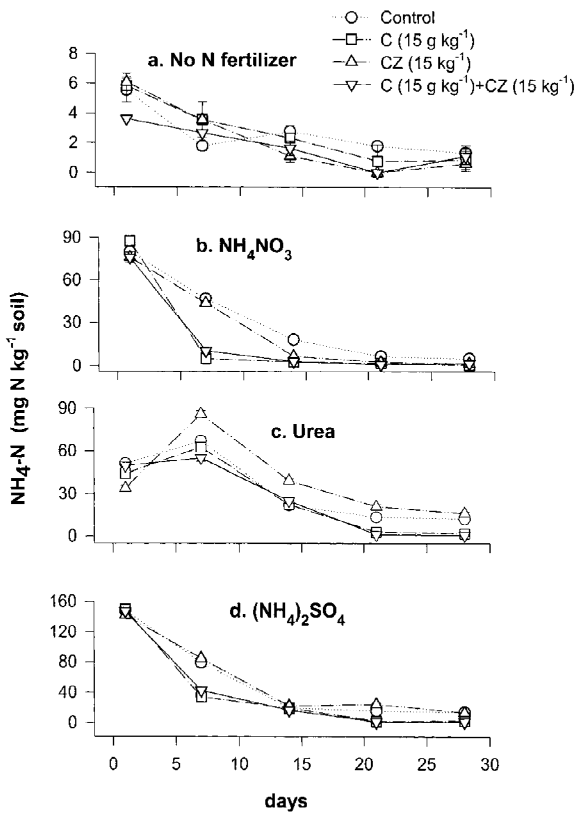

Figure 2. Dynamics of NH_4–N concentrations in a Riviera fine sandy soil amended with cellulose (C) and clinoptilolite zeolite (CZ) for different nitrogen sources (error bar=standard error).

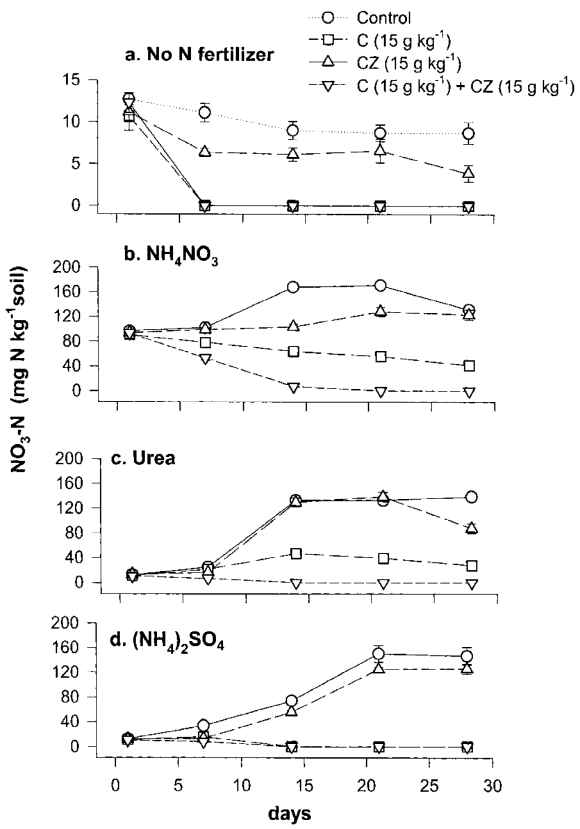

Figure 3. Dynamics of NO_3–N concentrations in a Riviera fine sandy soil amended with cellulose (C) and clinoptilolite zeolite (CZ) for different nitrogen sources (error bar=standard error).

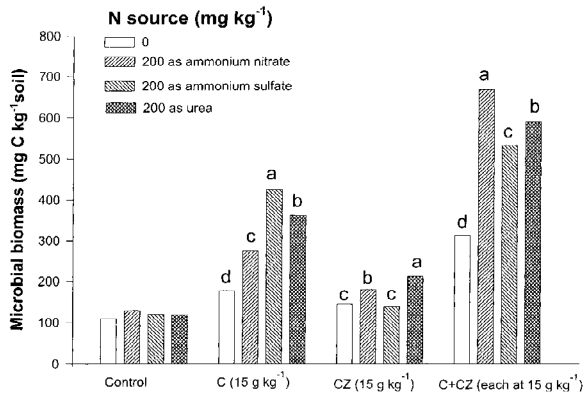

Figure 4. Microbial biomass in a Riviera fine sand with application of different nitrogen sources at the end of the 28-day incubation (C=cellulose, CZ=clinoptilolite zeolite, different letter on the top of each bar within the same group indicates significant difference at $P < 0.05$).

retention of NH_4^+ on the CZ, which decreased the availability of NH_4^+ for nitrification. The NO_3–N concentrations in the soil amended with cellulose were generally low (Figure 3), possibly due to increased N immobilization by microbial biomass stimulated by the newly added C source (Figure 4). The combined amendment of cellulose and CZ resulted in the lowest NO_3–N concentration in the AN- and urea-amended soil throughout the incubation after day 7 (Figure 3). The mechanism for the marked interaction effects of cellulose and CZ on NO_3–N concentrations in the soil is not fully understood. Perhaps CZ amendment increased soil pH and NH_4^+ retention by the soil, which could have favored microbial growth, thus, further increased N immobilization compared to cellulose application alone.

Microbial biomass at the end of the incubation

The microbial biomass in the soil without cellulose or CZ amendment was in the range of 100–120 mg C kg^{-1} (Figure 4), which is relatively low compared with an average value of 300 mg C kg^{-1} for most arable soils (Smith and Paul, 1991). This is probably due to limited nutrient and C sources of the sandy

Table 2. Statistical analysis of soil amendment effects on microbial biomass in soil

Treatments[a]	Zero N	AN	AS	U
Control	NS[b]	NS	NS	NS
C (15 mg kg^{-1})	*	**	**	**
CZ (15 mg kg^{-1})	*	*	*	*
C (15 mg kg^{-1})+ CZ (15 mg kg^{-1})	***	***	***	***

[a]AN – ammonium nitrate, AS – ammonium sulfate, U – urea, at 200 mg kg^{-1} soil; C=cellulose, and CZ= clinoptilolite zeolite.
[b]NS, not significant and *, ** and *** represent 5, 1 and 0.1% significant levels, respectively.

soil, which are not favorable to microbial growth. Addition of cellulose increased the microbial biomass C by 0.6, 1.5, 2.9 and 2.3 fold, in the soil with no N, AN, AS or U, respectively. There was a positive influence of N fertilizer application on microbial biomass C (Table 2), indicating that N might become a limiting factor for microbial growth in this soil after addition of organic C. Application of cellulose substantially increased microbial activity, thus enhancing transformation of applied N in soils (Figure 4). Microorganisms require N for their biomass growth with an average C/N ratio of 15. A large proportion of mineral N was probably used for the marked increase in microbial biomass in the cellulose-amended soil. The incorporation of mineral N into microbial biomass and other organic fractions might explain the reduced NH_3 volatilization and low concentrations of NH_4–N and NO_3–N in the cellulose-amended soils with application of 200 mg N kg^{-1} (Figures 1–3).

Addition of CZ also increased microbial biomass in the soils for all treatments (Figure 4). This could be attributed to improved availability of NH_4–N and other nutrients, which was favorable for the growth of microorganisms. Furthermore, CZ addition increased retention of NH_4^+ in the soil since it provides ion-exchange sites.

Application of CZ together with cellulose was more effective in increasing microbial biomass than either CZ or cellulose alone (Figure 4). The marked increase in microbial biomass by the amendment of cellulose plus CZ was in agreement with the significant decrease in NH_3 volatilization and reduction in concentrations of NH_4–N and NO_3–N in the soil (Table 1, Figure 1–3). These results suggest that soil amendment with cellulose or CZ is a potentially effective approach to reduce N losses in sandy soils by enhancing incorporation of mineral N into the microbial biomass.

Discussion

Nitrogen fertilization is essential for sustainable production of citrus. Citrus trees are mainly grown in sandy soils in Florida, which have a very low nutrient holding capacity because of low ion exchange capacity and organic matter concentrations. Moreover, because of the fluctuating water table, citrus in the coastal regions of Florida has a rather shallow rooting zone with most roots growing within 0–40 cm of the soil depth (Zhang et al., 1996). Therefore, N applied to the sandy soils that is not absorbed by the trees is subjected to leaching loss (Alva et al., 1998).

To overcome the leaching problems, fertilizers are generally applied to the citrus groves in February, May and October during the dry season (from mid-October to mid-June next year), and the major way of fertilization is dry application of water soluble granular using a mechanical spreader. The dry soil condition and high surface soil temperature in May or October enhance NH_3 volatilization from the surface-applied fertilizers, especially for the calcareous soils. The loss of N via NH_3 volatilization from NH_4NO_3 and polymer coated urea ranged from 80 to 180 g kg^{-1} under field conditions (Paramasivam et al., 1999). Ammonia volatilization not only results in increased production costs, but also raises concern regarding N contamination of surface water and groundwater (Davies, 1996).

Since 1994, many studies have been conducted to develop nitrogen best management practices (BMPs) to reduce N losses via leaching or volatilization and to increase N utilization efficiency (Alva and Paramasivam, 1998; Alva et al., 1998). Soil remediation is an integrated part of the N BMPs program. Clinoptilolite zeolite has been used to reduce NH_3 emission from farm manures (Amon et al., 1997) and as NH_4^+-loaded exchange fertilizer (Perrin et al., 1998a,b) because of its high CEC. However, few studies have been conducted to evaluate CZ as a soil amendment for calcareous sandy soils to reduce N losses. Organic materials such as composts have been increasingly used as mulch in citrus groves, but the mechanisms responsible for beneficial effects of organic amendment remain to be unclear. The results from this study demonstrate that addition of CZ and/or cellulose is potentially effective in reducing NH_3 volatilization and NO_3–N concentration in a calcareous Riviera fine sand. The mechanisms involved include: (1) increased NH_4^+ retention on cation-exchange sites provided by the CZ; and (2) enhanced microbial growth and activities. As a result, more surplus N is incorporated into the expanded microbial biomass. The microbial biomass N, however, is eventually available to the plants (Smith and Paul, 1991). In the calcareous sandy soil, the applied N (NH_4–N or NO_3–N) that is not absorbed by the plant is subjected to losses through volatilization or leaching. Therefore, the incorporation of mineral N into organic fractions that are less subjected to loss and potentially available to the plants could be considered as a beneficial process. The positive effects of interactions between the CZ and organic carbon such as cellulose on reduced N losses in calcareous sandy soils merit further attention.

It must be pointed out that under field conditions, since fertilizers are mainly surface applied in citrus production system, effects of zeolite/organic C on reduced NH_3 volatilization may not be as pronounced as what we obtained from the incubation study in which fertilizer was incorporated into soil. However, it is common practice in Florida to apply irrigation for 0.5–1 h (using an under the tree microirrigation system with one emitter per tree) to dissolve the fertilizer into the surface soil after it is applied. Therefore, a significant reduction in NH_3 volatilization by soil amendment of zeolite/organic C could be still expected.

Farms and manufacturers in the USA produce annually more than one billion tons of by-products including ground CZ and bioavailable C sources such as paper mill wastes (Karlen et al., 1995). The availability of amendment materials may not be a limiting factor. However, transportation and application costs may affect the potential use of CZ in fields where cheaper CZ sources are not locally available. On the other hand, organic materials can be either a sink or a source for N, depending on the C/N ratios of organic materials and the soil. Moreover, the timing of N assimilation and mineralization with the fertilization and demand of plant growth is a complicated issue. Therefore, for beneficial use of these materials to improve sandy soils and reduce N loss in the citrus production system, some long-term field study needs to be conducted to define a number of factors such as CZ quality, C/N ratio of organic materials, and timing of soil amendment and fertilization with nutrient demand of citrus trees, etc.

In addition, it would be more convenient to amend the sandy soil with zeolite at planting since this material has relatively long-lasting effect and soil incorporation would be more difficult after the trees are grown up. Based on this study, 15 g zeolite per kg soil is needed to effectively reduce N losses in the sandy soil. If soil incorporation of zeolite is limited to the top 10 cm (which should be sufficient to catch NH_4^+ dissolved from the surface applied fertilizers), then 22.5 tons of zeolite will be needed per ha of land. As an alternative approach, zeolite/organic C may be also applied together with chemical fertilizer N to reduce N loss and potential contamination to waters. Further study is needed to explore this aspect of zeolite/organic C application for sandy soils.

Conclusions

Soil amendment with organic carbon such as cellulose, clinoptilolite zeolite (CZ), or both has a potential to reduce N losses via NH_3 volatilization and NO_3 leaching in calcareous sandy soils. The mechanisms responsible for the beneficial effects of the amendments are involved with increased soil retention capacity for NH_4^+ and enhanced microbial assimilation of available N in the soil. However, many factors such as availability, quality, and costs of CZ, quality of organic matter, and timing soil N availability with demand of the citrus trees need to be carefully considered to explore the beneficial use of the amendments in citrus groves. Some long-term studies are needed to develop soil amendment strategies for reducing N losses in sandy soils under field conditions.

Acknowledgment

This study was supported, in part, by a grant from the Florida Department of Agriculture and Consumer Services, Tallahassee, Florida.

Florida Agricultural Experiment Station Journal Series Number R-07003.

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Section editor: Z. Rengel